

GB00/2059

The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ

4

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears a correction, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

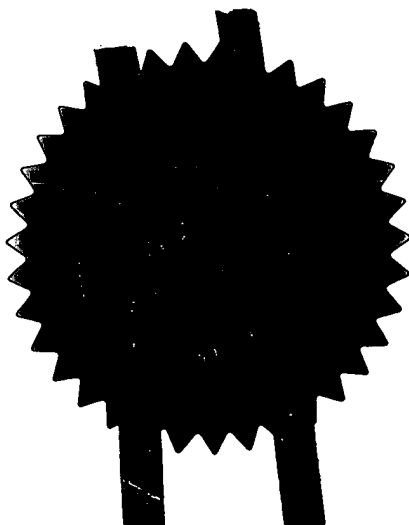
In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

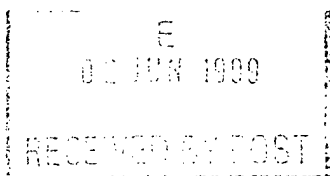
Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

**PRIORITY
DOCUMENT**
SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

Signed 

Dated 13 June 2000





Patent Office

02JUN99 E451231-1 002973
P01/7700 0.00 - 9912694.8

Request for grant of a patent

The Patent Office
Cardiff Road
Newport
Gwent NP9 1RH

1	Your reference	MRH/P32052		
2	Patent application number	02 JUN 1999 9912694.8		
3	Full name, address and postcode of the applicant	Bain, Peter Stewart 103 Rein Road, Wakefield WF3 1JQ, GB 7671787 001 Rdes MANFRE Mafrè, Giovanni via Vicenza 11, 37042 Caldiero, Verona, Italy 7671761 001 Rdes 07734239601 United Kingdom		
4	Title of the invention	Adhesive		
5	Name of agent Address for service	Harrison Goddard Foote Belmont House 20 Wood Lane Headingley Leeds LS6 2AE 14571001 ✓		
6	Priority applications	Country	Priority App No	Date of Filing

Patents Form 1/77

Page 1 of 2

7	Parent application (eg Divisional)	Earlier Application No	Date of Filing
8	Statement of Inventorship Needed?		
9	Number of sheets for any of the following (not counting copies of same document) Continuation sheets of this form Description Claims Abstract Drawings	8	
10	Number of other documents attached Priority documents Translations of priority documents P7/77 P9/77 P10/77 Other documents		
11	I/We request the grant of a patent on the basis of this application. <div style="text-align: right;"> Signature <u>Michael R Harrison</u> Date 1 Jun 1999 </div>		
12	Name and daytime telephone number of person to contact in the United Kingdom <div style="text-align: right;"> Michael R Harrison +44 113 2258350 </div>		

Adhesive

The present invention relates to an adhesive for use in sealing together two surfaces, for use especially in the glazing industry in general and in securing vehicle
5 windscreens and/or windows and/or other vehicle features that may require replacement such as, without limitation, car panels; the invention also provides a method of use for installing and/or replacing vehicle windscreens and/or other fixed glazing on vehicles.

10 Background of the Invention

Typically to install a window pane in a wooden/plastic/metal frame, the glass pane is firstly held in position against nails or other clasps and then fixed into position by putty or plasters material. Conventional putty is a cement made from whiting and
15 linseed oil which hardens over time to provide a peripheral rim of the window pane, thus separating interior and exterior environments and preventing air, moisture and/or heat transfer. The installation is completed once the putty has dried and this usually takes up to 6 hours or so depending on the kind of plasters used.

20 To remove a window pane after it has been fixed in position in a frame requires the window itself to be shattered so that the hardened putty or plasters can be scraped/chiselled away from the frame. The removal operation can cause damage to the frame and varnishes.

25 In use, the window pane is held rigidly around its edges so that even relatively small vibrational mechanical movements such as with earthquakes or bomb blasts or strong winds can cause the window pane to shatter.

In the automotive industry, cars direct from the factory production line typically have
30 the windscreens and other fixed windows, including light assemblies fixed into position by placing the glass against a frame rim and using adhesives so as to direct

glaze the glass. The life span of a windscreen and other fixed windows are significantly shorter than that of the vehicle itself partially due to degradation or damage or being deliberately broken by vandals/car thieves. Thus a motorist may need to replace the windscreen several times during the vehicle's lifetime.

5

Additionally, glued glazings have to be replaced any time the window, especially the windscreen, has been damaged in its optical performance by, for example, impact stones or other fractures or abrasion by wipers. Damage to the window surface can increase the scattering of light and may reduce the visibility to levels below safety limits. Moreover, regulations of motor worthiness (MOT) stipulate that there can be no chips or visual impairments on laminated windscreens, so whereas recent improvements have made the windscreens shatter-proof, they are still prone to chipping and fracturing and thus will require replacement.

15 The process of replacing vehicle windscreens is both laborious and time consuming. The automotive glass fitter has first to remove the defective windscreen (usually in intact form), however the windscreen is firmly bonded in place and the adhesive sealant is hardened. Typically the fitter uses a device comprising a cheesewire. The cheesewire is used to cut/saw through the hardened rubber along the periphery of the windscreen. This process requires physical force and can lead to musculo-skeletal conditions in the fitters themselves as a result of repetitive strain injury. Further problems associated with this method are that the cheesewires can overheat due to friction, additionally the wires themselves can break.

25 Other methods of detaching the windscreen from the adhesive sealant include: the use of mechanical oscillator knives/cutters to cut through the hardened material or; directed heat such as a laser beam to soften the sealant prior to removing the windscreen with either cheesewire or specialised bladed tools. The problem with a method where heat is directly applied to the sealant is that the heat required to soften the hardened adhesive sealant can concomitantly and inadvertently damage the vehicle's paintwork and/or other exterior surfaces. For example, a pulsed laser that is

set to pulse too fast will not generate enough energy to char the adhesive sealant and a pulsed laser that is set too slow will burn the adhesive sealant and liquify it.

Once the windscreen has been freed from the rubber sealant it can be removed and the surround scraped before it is replaced. It is known from the prior art to use urethane based adhesives to fix/seal the replaced windscreen in place and to apply the adhesive from a dispenser gun to specific peripheral edges so as not to impinge on the viewing capacity of the windscreen. The adhesive typically takes about 8 hours to cure.

10

Recent advances to the industry have provided for the inclusion of fast cure agents/catalysts so as to speed up the time from vehicle drop-off to vehicle collection. The fast cure agents/catalysts can be provided pre-mixed in the adhesive composition or alternatively can be mixed with the adhesive at the point of exit from a dispensing gun. However the problem still remains that the removal of a defective windscreen and its subsequent replacement is a laborious and time consuming process which can result in damage to the dashboard interior or vehicle paint-work.

An adhesive that could satisfy vehicle safety crush and crash standards and provide for easy, effective and damage-proof removal of a defective windscreen or other fixed glazing from a vehicle would offer immediate improvement to the industry and consumer.

In a completely different technical field it is known to provide thermoexpandable microcapsules or microspheres for use in the manufacture of porous or lightweight materials with acoustic and thermal insulating properties as covering materials or walls. The microcapsules or microcapsules comprise a polymer shell of certain thickness and mechanical properties. The shell encapsulates materials such as volatile organic solvents, expandable gases or activating agents or any other such material which is capable of interacting at certain specified conditions with a selected matrix. The microcapsules, when heated to a sufficient temperature, typically in the

region of about 75-180 ° C. depending on the encapsulated substance, can produce an increase of their volume at high expansion rate in some instances at a volumetric expansion limit of up to 70 x original volume. The percentage of the microspheres in a given composition, their expansion ratio, the temperature operating range, the softening transition range of the matrix are all parameters which are able to influence the expansion volume of a layer between two materials.

We have discovered that by mixing a powdered form of specially developed microcapsules with an appropriate adhesive that the resulting composition is effective at providing glazing adhesion and sealing. Moreover, of particular advantage is that the adhesive bonds in the composition can be weakened by the application of direct heat to the composition thereby allowing sufficient softening of the adhesive material so that a vehicle windscreen/fixed glazing can be easily and rapidly removed.

We believe that the invention provides the first application/use of a polyester material in the automotive glazing industry.

It will be appreciated that the adhesive of the invention has application in other areas especially where two surfaces are to be bonded together and where one surface may subsequently need replacing following damage or wear, for example, and without limitation; shower doors and vehicle panels.

Reference herein to vehicle is intended to include, without limitation car, lorry, van ship, boat, plane, cable car, helicopter, hovercraft and any other form of transport in which there is fixed glazing.

Statement of the Invention

In its broadest aspect the invention provides a composition comprising an adhesive together with a heat triggered adhesion-deactivating microcapsule or microsphere.

According to a first aspect of the invention there is provided a composition for use as a glazing adhesive comprising an adhesive agent and dispersed therein thermoexpandable microcapsules, the microcapsules each comprising a polymer shell
5 which encapsulates at least one expanding gas or volatile expanding agent.

Preferably, the adhesive agent is a urethane or polyurethane or polyvinylchloride or a MS polymer.

10 Preferably, the thermoexpandable capsules are microspheres or hollow fibres in the form of a powder.

The powder can be provided with the adhesive agent in a pre-mixed form in a container or the powder can be introduced into and mixed with the adhesive at a
15 point of exit from a dispensing device. The dispensing device can be followed by a static mixer suitably lubricated at the point of exit of the composition for optimal blending.

Preferably, the composition additionally comprises a fast cure agent or catalyst,
20 whereby the adhesive composition is rapidly cured or set.

Preferably, the composition comprises a colouring agent so that the cured composition is black.

25 Reference herein to cure is intended to mean the hardening or setting of the adhesive mixture, the hardening or setting can be either chemically or non-chemically enhanced.

Preferably, the microcapsules encapsulate more than one material, ideally the
30 material is selected from the group consisting of an expanding agent, an agent capable of sublimation, water or an activator agent.

Preferably, the activator agents are capable of foaming or of shrinkage. The present invention includes the simultaneous use of microcapsules encapsulating a variety of different agents, either separately or in combination. The additional microcapsules are activated by the breaking or permeation of the polymer shell whereby their contents are released so as to interact with the adhesive mix. Their activation is as a result of specified applied conditions and thus is controllable. Microspheres do not break/fracture their shells in the expanding state maintaining their integrity, so that an activated composition comprises intact expanded microspheres and microcapsules which have released their contents into the composition.

It will be appreciated that the expanding agent inside the capsule is capable of activating a foaming process of the adhesive composition and that the agent capable of sublimation is to allow the composition to expand under certain specified conditions. Both these processes will occur after the breakage/fracture of the microcapsule shell and thus contribute to facilitating the removal of fixed glazing. The inclusion of water in the microcapsules is to allow the adhesive composition to weaken in certain conditions and the presence of an activator agent is to crosslink or polymerise the adhesive composition whereby shrinkage occurs and the adhesive composition weakens.

Preferably, the microsphere's diameter is in the range 10 to 120 μm .

Preferably, the microcapsule shell thickness is in the range 3 to 7 μm .

Preferably, the composition comprises microcapsules in the range of 1-30% by volume, and more preferably in the range of 10-15% by volume.

Preferably, the composition is activated by heat wherein the heat activation range is 80-160° C and ideally 120-125° C.

The temperature of a vehicle windscreen can reach over 100°C in natural soak conditions. Thus the heat activation of the microcapsules in the composition of the invention needs to be in excess of any natural temperatures that may occur whilst the heat activation must be below those that could damage a vehicle dashboard trim or paint-work. It is envisaged that the composition of the invention will have application in many fields and many diverse climates hence the composition may be provided with selected heat activation ranges depending on its intended use and/or country of use for both kinds of microcapsule: the expanding microspheres and the microcapsules with breakable shells. For example, in the instance of the composition being used for fixing and sealing a shower door it is envisaged that the adhesive composition microcapsule heat activation range would be in the region of 80-100 °C, whilst for the majority of automotive glazing the microcapsule heat activation range would be in the region of 120-125° C. Optionally in either composition a user may require microcapsules capable of releasing a curing agent and/or an activator agent capable of shrinkage.

Heat activation of the microcapsules causes the beads/fibres to thermoexpand thus creating pressure along the rim of glazing. This in effect reduces the viscosity of the adhesive material and expands the material as a whole with the result that the adhesion of the glazing is loosened and the windscreen can be easily removed.

According to a second aspect of the invention there is provided a method of installing and/or replacing a vehicle windscreen or fixed glazing comprising the steps of:

- (i) placing a windscreen flush against a window aperture rim of a vehicle,
- (ii) dispensing the composition of the first aspect of the invention around the periphery of the windscreen,
- (iii) allowing sufficient time for the adhesive to cure,
- (iv) applying a heat source to the cured adhesive wherein the heat applied is sufficient to cause thermoexpansion of the microcapsules and thus weaken the adhesive bonds of the composition, and
- (v) removing the windscreen from the main vehicle body.

It will be appreciated that the method of installation involves steps i-iii whilst the method of replacement involves steps iv-v.

5 By using the method of the invention as herein described, a vehicle windscreen or fixed glazing can be removed and/or replaced more rapidly and with less damage to the frame and/or paint work than by prior art methods, thus the method is more cost effective to both the fitter and customer.

10

P32052.2